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FINDING THE POROSITY PERECENTAGE FOR SMART ALLOY (Cu-Al-Ni) SAMPLES WHEN CHANGING THE SINTERING TIME AT THE FIRST STAGE

AHMED ABDULRASOOL AHMED ALKHAFAJI

Assistant Professor, Department of Mechanical Engineering, College of Engineering - University of Baghdad, Baghdad, Iraq

ABSTRACT

In this research the effect of porosity is studied at three different holding times(1.5, 1 and 0.5 hours) for the first sintering stage, to fixing aluminium in its solid state phase at temperatures (500°C), of the memory alloy based on copper. The powder metallurgy method has been used to manufacture 3 samples for this alloy with(83%Cu,13%Al,4 %Ni). To make sure of the generation phases for Marten site and Austenite, physical tests (Optical microscopy, XRD, and SEM) were carried out before and after sintering.

The porosity testing was conducted on these samples, depending on the specification (ASTM B 328). The percentage of porosity is generated at the three holding times for the first stage was calculated, according to the equation adopted that.

The results give, the lowest porosity% was at 0.5 hours with a value of 2.907 %, at 1.5 hours was the highest value for porosity 3.733%, at either hour the value of porosity was 3.720%. In this way we observe the effect of the holding time of the first stage of sintering on the porosity. The relationship was a positive, the greater the holding time, it gives a greater percentage of porosity. It gives an indication of the utilization of those in the process of sintering to obtain the required percentage of porosity, especially in products that need it, such as self-lubricating bearings.

Keywords: (Cu-Al-Ni) Smart Alloys, Powder Metallurgy, Porosity, XRD & SEM

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Original Article

INTRODUCTION

Copper based alloys exhibit shape memory (smart material). Alloys within a certain range of composition, the shape memory Alloys (SMAs) has two phases: These two phases occur diffusion, less the solid phase obtained during cooling is called Martensite or low temperature phase. The parent phase in which the transformation occurs is called the Austenite, which is known as high temperature phase [1, 2]. A smart material is a term used to describe material that has an ability to adjust its properties and shape due to changes in applied stress and or temperature. There is more than one application make smart materials or smart alloy very importance like actuators or piping fastener's or medical applications [3]. Cu-Al-Ni alloy get attention as smart alloy because its ability to work fine at high temperature about 200^{o}C [4]. Pores in PM are described as the spaces between particles in the powder which are two types: the first is called open pores, it's observed on the surface while the second type is called close pores which are not open to the outside and these pores are less through compacting and sintering

process [5].

Ahmed, and Sarah [6] Porosity have been studied, the samples manufacturing smart alloy composed of copper alloy, aluminum and nickel. The mixing materials weight percentage is 83% wt Cu - 13% wtAl-4% wt Ni. Has been followed by the compacting process. The powder compacted with three different values of the pressure (300, 500, 700)MPa. The sintering process has been in a tube and vacuum furnace sintering environment at three different values of sintering temperature values (700, 800, 900)^{o}C. The measurement and calculation for density & porosity shown highest average density (5.583g/cm) at Sintering temperature (900^{o}C) and Compacting load (300MPa).

Ahmed and Hasan [7] Investigation of relationship between shape memory effect and interconnection porosity under the multiple sintering time of smart alloy Cu-Al-Ni. In this study the powder metallurgical method has been used to manufacture Cu83%-13%Al-4%Ni alloy by producing 5 samples every sample sintered in difference sintering time(3, 4, 5, 6, 7) hours. The samples also heat treated to stabilize Marten site phase. The result of shape recovery and porosity testing analyses by using artificial neural network predicting system to predict shape recovery and porosity behavior between three and seven sintering hours with smaller time step Due to there is no physical relationship between porosity and shape recovery. The relationship between porosity and shape effect curved by using excel program taking the result from predicting system of artificial neural network. The curves show a direct relationship between shape recovery and porosity with respect to time.

Ahmed and Bassam [8] Study the effect of Cu-Al proportions in smart (Cu-Al-Ni) alloy for better mechanical properties by using artificial intelligent. In this work study effect two elements (Cu, AL) of the alloy (Cu-Al- Ni) on the physical and mechanical properties which is considered one of the smart materials. This alloy has a standard weight percentage in [83%Cu-13%Al-4%Ni]. Selecting four different weight percentages of elements (Cu-Al) include [78%Cu, 18%Al], [80%Cu, 16%Al] [82%Cu, 14%Al] and [84%Cu, 12%Al]which manufactured by powder metallurgy technique with a constant weight percentage of element Ni in each the percentages. One conclusion was, increase Al% content out of range (12-14)% lead into increase amount porosity in the alloy which reach into 25.9% in weight percentage [78%Cu-18%Al-4%Ni] although the compacting pressure is constant (650MPa) in all the weight percentages.

Taher and Ahmed [9]. Study the effect of (Al-Ni) & (Cu-Ni) concentration ratios on the hardness and porosity of ternary (Cu-Al-Ni) smart alloys. In this research, the newly achieved results determined the best ratio of (Cu-Ni) and (Al-Ni). Porosity testing in accordance to the ASTM B328-(1996) so as to show the effects of (Cu-Ni) & (Al-Ni) concentration ratios on hardness & porosity of (Cu-Al-Ni) smart alloys. The analysis results proved that when there is an increase in Al and Ni concentration in the alloy, lead, it will automatically increase the hardness and porosity, but the increase in Al ratio shows more effect than the increase in Ni ratio.

The apparent porosity can be determined from Equation. 1 [10]:

$$P= [(B-A) * 100/ (B-F)* Do] * D_w \quad (1)$$

P = interconnecting porosity by volume, %.

A = mass in air of oil-free sample, g.

B = mass of oil-impregnated sample, g.

F = mass of oil impregnated specimen in water with mass of wire tared, g.

Dw =density of water at the immersion temperature, (0.9984 g/cm^3 at 19°C).

Do =density of the oil, 0.88 g/cm^3.

This research focuses on the change of time in the first stage of sintering at a temperature of 500^{o}C from the porosity of this type memory alloy. It is through our reading of previous research that we did not get the research stopped at this point, Figure 1 showing diagram of sintering procedure.

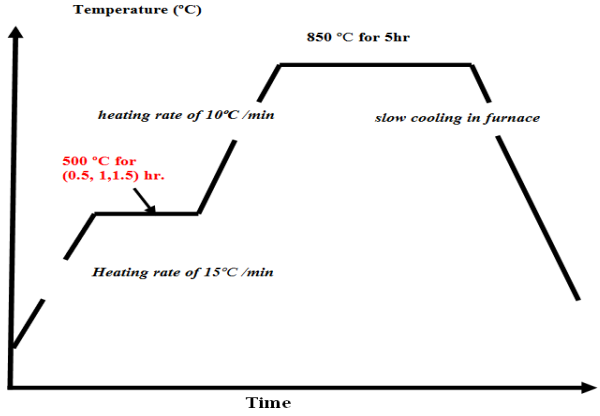


Figure 1: Diagram of Sintering Procedure

EXPERIMENTAL WORK

The experimental work of this research is similar to previous research, mentioned in the references or in the methods of manufacturing Powder Metallurgy. The difference here is in the time of sintering of the first stage, special in keeping the Aluminum in a solid state, before heating to the second stage of sintering 850^{o}C, see figure 1. The steps, stages and tests between the different stages are as follows.

Powder Preparation

Cu-Al-Ni was prepared by powder metallurgy method. The powder which has been used in this study was brought from (Sky spring Nan materials, USA) with a purity of 99% and an average particle size of 45 microns (-325 mesh). The first step was to mix the powder of Cu83%-Al13%-Ni4% percentage by horizontal drum mixing with 78 rpm speed for 6 hrs with 1% acetone (by volume) to prevent particle separate ration due to different densities also acetone decreases the friction between particles Figure 2 shows the horizontal drum that was used for mixing the powder.

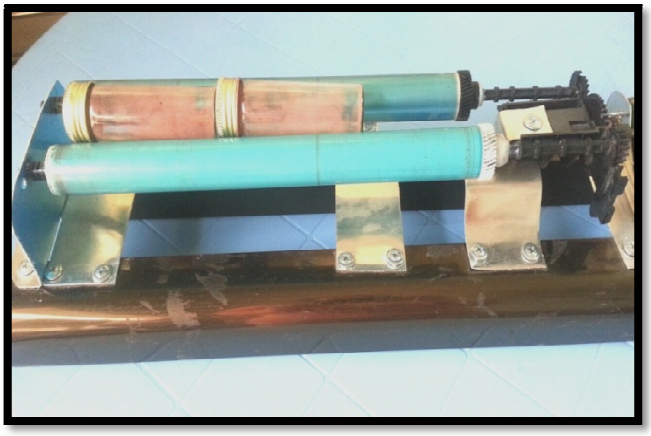


Figure 2: Horizontal Drum Mixing

Compacting

Powder mixture was compacted at 650 MPa using cylindrical die of 11 mm diameter. These sample dimensions are (11mm dia. x 16.5 mm length) Figure 3 shows that. The compacting process from two sides to increase the homogeneity of density along the sample. After compacting finish the punch put on hold at 650 Mpa for 2 minutes to prevent the spring back of the sample Figure 4 as shown. In this research, Figure 5 shows three samples that were manufactured. The optical microscopy with 200x was then used to investigate the microstructure of the samples after compacting, as shown in the Figures 6, 7 and 8.

Figure 3: Die, Punch and Holder for Mold

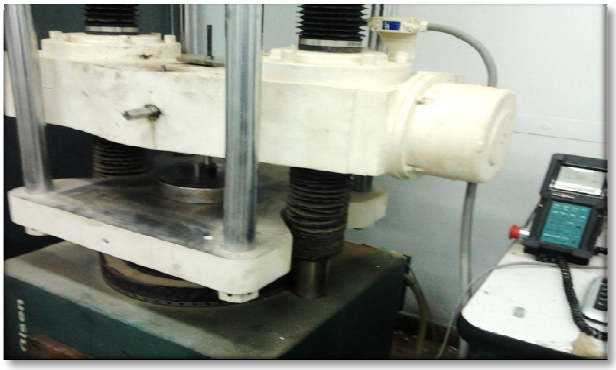


Figure 4: Uniaxial Press Machine

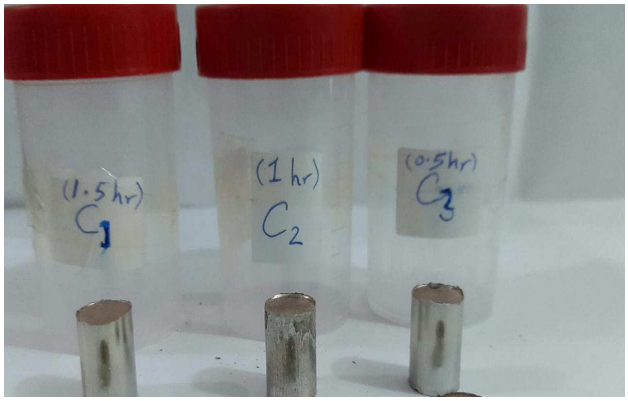


Figure 5: Samples Manufacturing

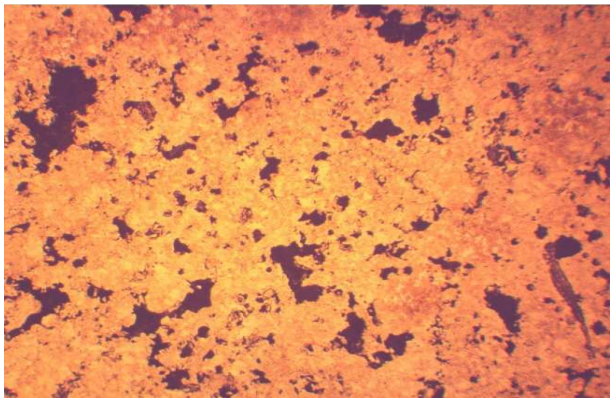


Figure 6: Microstructure of Sample C1 after Compacting

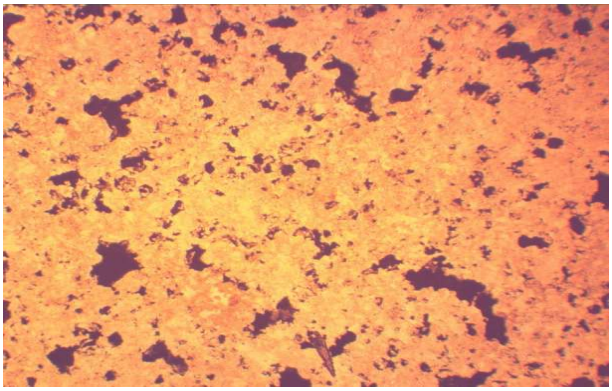


Figure 7: Microstructure of Sample C2 after Compacting

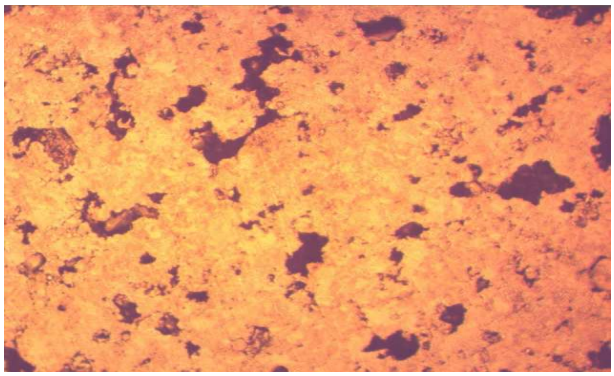


Figure 8: Microstructure of Sample C3 after Compacting

Sintering

After the compacted samples were then sintered in two stages. Samples divided in three to be sintered in the first stage at 50^{o}C, heating rate of 15^{o}C/min from room temperature. Different time of these samples is holding it this stage (0.5, 1 and 1.5) hr respectively. The second stage for all have the same condition of sintering at 850^{o}C hold it 5 hr, for every sample directly after the first stage, heating rate (10^{o}C / min.) to reach this after the first stage, Figure 1 shows the diagram of sintering procedure. Then samples were left to cool in furnace to room temperature for the purpose of bonding particles of the sample in the solid state. The sintering process was done an electrical tube furnace with a vacuum atmosphere to prevent oxidization of the samples, Figure 9 shows the furnace and Figure 10 shows the samples after sintering.

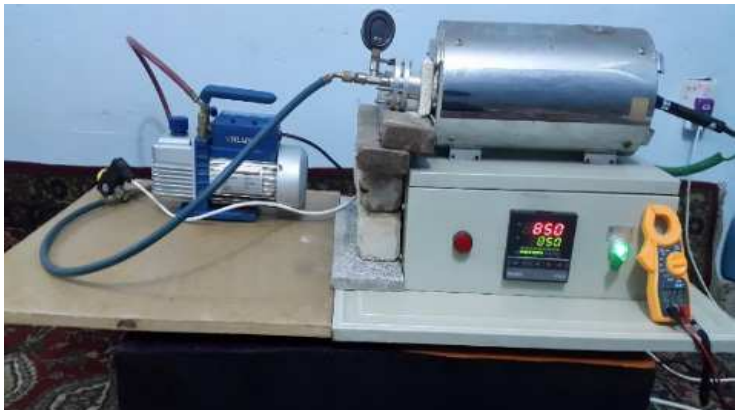


Figure 9: Electrical Furnace with a Vacuum System

Figure 10: Samples After Sintered

Heat Treatment Procedure

By the same system (electrical tube furnace with a vacuum atmosphere to prevent oxidization of the samples) was used for all samples. To transform the Austenite samples after sintering to Marten site, two stages of heat treatment were employed. The first stage (aging the samples at 800^{o}C hold it at one hour, and after that rapidly quenched in iced water) and the second stage (heating the samples to 100^{o}C and hold it for two hours and left it to cool in furnace).

TESTS AFTER MANUFACTURING

After etching the samples so as to prepare them for microscopic inspection the following tests were performed

Physical Tests

Optical microscopy Figures 11, 12 and13 show the microstructure of samples (C1, C2 and C3) after sintering.

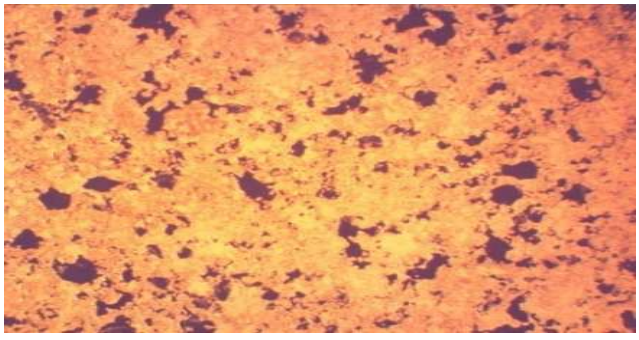


Figure 11: Microstructure of Sample (C1) After Sintering

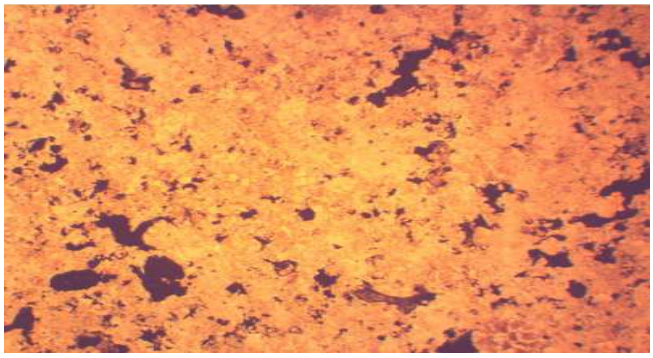


Figure 12: Microstructure of Sample (C2) After Sintering

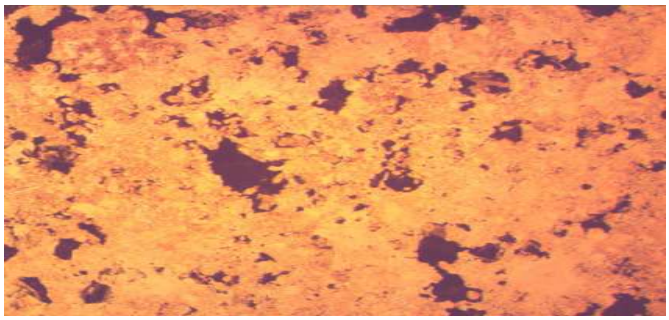
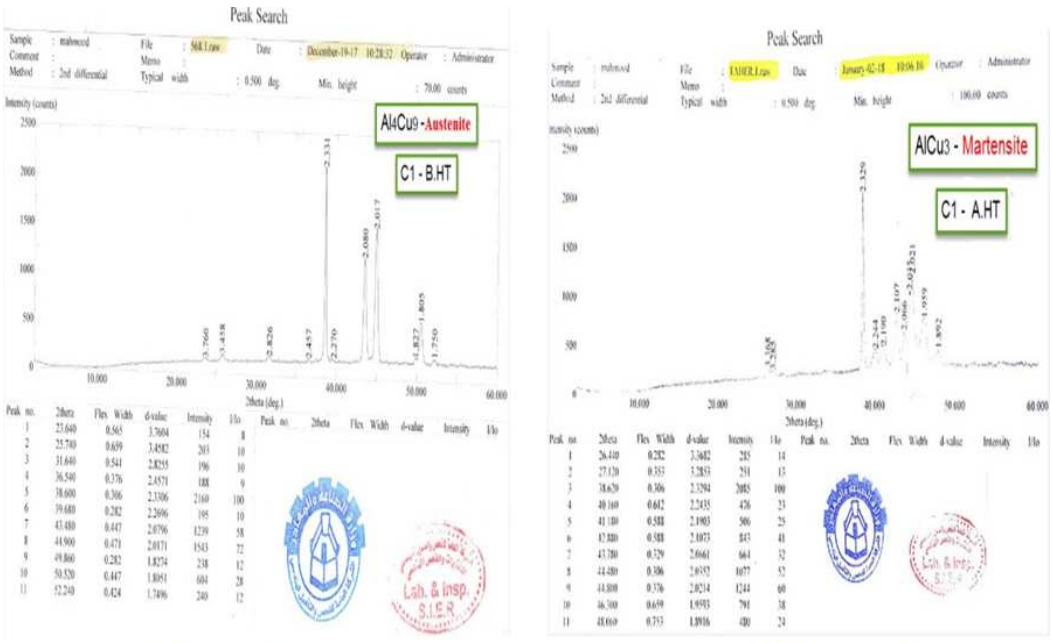


Figure 13: Microstructure of Sample (C3) After Sintering

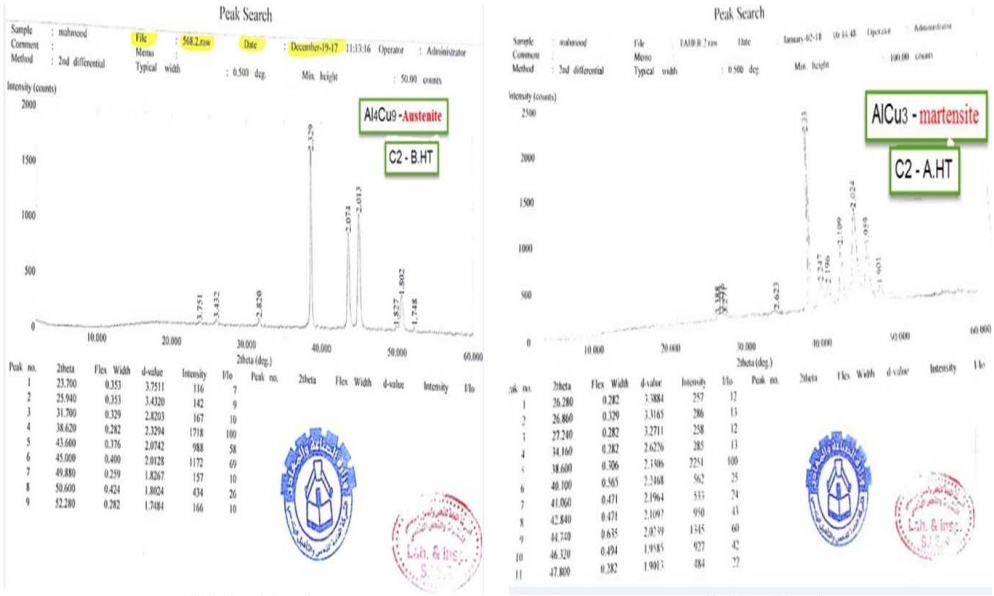
XRD

X-ray diffraction tests were done on samples (C1, C2 andC3) before and after sintering, to cover the range of changes in first stage sintering time. The purpose of mentioned test was for investigating the effect of sintering time on Martensite transformation because the martensite transformation is the most important step to ensure that the sample is a smart alloy. Figures 16, 17 and 18 show the results of these tests.



C1 Before Sintering **C1 After Sintering**

Figure 14: XRD Tests Before and After Sintering for Sample C1



C2 Before Sintering C2 After Sintering

Figure 15: XRD Tests Before and After Sintering for Sample C2

C3 Before Sintering C3 After Sintering

Figure 16: XRD Tests Before and After Sintering for Sample C3

SEM

Scanning electron microscope was used to obtain clear observation on microstructure to see the Martensite phase as shown in Figures 17, 18 and 19.

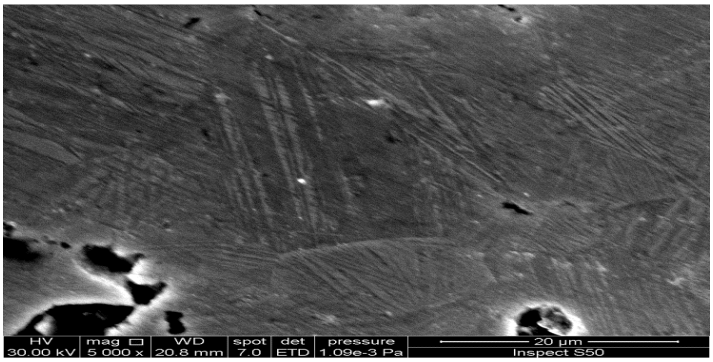


Figure 17: SEM after Sintering Sample C1

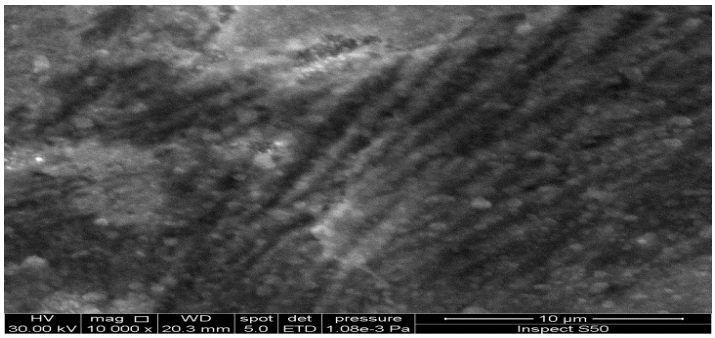


Figure 18: SEM after Sintering Sample C2

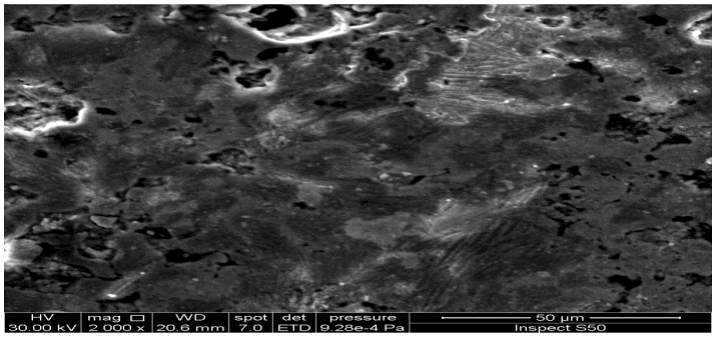


Figure 19: SEM after Sintering Sample C3

Porosity Tests

Conduct porosity testing on samples, using sensitive balance (4-digits), to weigh each weight percentage of samples in three different cases.

- Should be weighted in the air.
- When it is immersed in the closed container that contains oil, and using a vacuum system for 30 min inside the closed container.
- Through opening the container and leaving it aside for 10 minutes, after which it has to be weighed in air after cleaning the sample from excess oil. In the third case, the oil saturated sample should be weighed in water using bin with the hock. The apparent porosity can be determined from equation 1. Table 1 shows the results and calculations when applying the equation for porosity calculation for the samples under study.

Table 1: The Results and Calculations, % for Porosity

Samp. No.	A (g) in air	B (g) in oil	F (g) in water	Porosity%
C_1	2.6674	2.7196	1.1334	3.733
C_2	2.5923	2.6438	1.0728	3.720
C_3	2.7428	2.7835	1.1951	2.907

RESULTS AND DISCUSSIONS

The compacting efficiency shown in the Figures 6, 7 and 8 for the optical microscopy testing, the distribution and homogeneity of alloying elements in all the samples, black spaces in the images indicates porosity. In the Figures 11,12 and 13 that show microstructure of samples after sintering, the black spaces were shrinking and decreased their number, increased the linkage between the elements. But there is a clear difference between samples, due to the different time of the first sintering.

Figures 14-19 show the XRD tests before and after sintering for samples and SEM after sintering samples. The purpose of these tests is to make sure that there are two phases (Austenite, Martensite) are generated of alloy.

The objective of the research is to know the effect of the sintering time of the first stage on the porosity. Table 1 explains the results of the porosity test and calculations. The lowest porosity% was at 0.5 hours with a value of 2.907%, at 1.5 hours was the highest value for porosity 3.733%, at either hour the value of porosity was 3.720%. In this way we observe the effect of the holding time of the first stage of sintering on the porosity.

CONCLUSIONS

The main conclusion of this research is to investigate the effect of time holding on the samples in the first stage of sintering on the percentage of porosity. The results of the study showed that there is effectively the impact of this time holding. The relationship was a positive, the greater the holding time, It gives a greater percentage of porosity. It gives an indication of the utilization of those in the process of sintering to obtain the required percentage of porosity, especially in products that need it, such as self-lubricating bearings.

The size of the generated porosity affects other mechanical properties, as earlier studies have shown on this type of SMA. Because the mechanical properties give a difference between them, Such as the percentage of recovery and hardness microstructure on this type of SMA, at this level of sintering and at the same time to keep.

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